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Re: Inventor(s): SONGLIN XU, ZHIWEN SUN, DRAGAN PODLESNIK, XUEYU QIAN
Title: NEW METHODOLOGIES TO REDUCE PROCESS SENSITIVITY TO THE CHAMBER CONDITION

Transmitted herewith is the patent application identified above, including:

- ☒ Specification, claims and abstract, totaling 28 pages.
☒ Drawings totaling 6 pages, ☒ Formal ☐ Informal.
☒ Executed Declaration and Power of Attorney.
☒ Information Disclosure Statement w/ Form 1449 and References.
☒ Assignment of the invention to **Applied Materials, Inc.**
☒ Assignment Recordation Cover Sheet



FEE CALCULATION					
Fee Items	Claims Filed	Included With Basic Fee	Extra Claims	Fee Rate	Total
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UNITED STATES PATENT APPLICATION FOR:

NEW METHODOLOGIES TO REDUCE PROCESS SENSITIVITY
TO THE CHAMBER CONDITION

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Certification Under 37 CFR 1.10

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NEW METHODOLOGIES TO REDUCE PROCESS SENSITIVITY TO THE CHAMBER CONDITION

BACKGROUND OF THE INVENTION

5 Field of the Invention

The present invention relates to an apparatus and method for plasma processing. More particularly, the invention relates to methods and apparatus for minimizing process sensitivity to chamber conditions during etching processes.

10 Background of the Related Art

In the fabrication of integrated circuits and other electronic devices, multiple layers of conducting, semiconducting and dielectric materials are deposited and removed from a substrate during the fabrication process. Substrate etching methods and apparatus used in device manufacture for the purpose of removing material from the substrate are well known. Typical etching techniques include wet and dry etching. However, wet etching is typically limited to fabrication of components with lateral dimensions of a micron or greater. Solid state devices and integrated circuits are now routinely fabricated with sub-micron or even nanometer scale components. Therefore, dry etching is now the preferred etching method.

One dry etching technique is commonly known as plasma enhanced etching ("plasma etching"). Plasma etching is very well suited for the manufacture of nanometer sized devices. A conventional plasma etching reactor includes a reactor chamber and an apparatus for producing a plasma within the reactor chamber. The plasma may be produced inductively, *e.g.*, using an inductive RF coil, and/or capacitively, *e.g.*, using a parallel plate glow discharge reactor. Typically, the plasma is struck and maintained both capacitively and inductively.

In general, plasma etching involves positioning a mask on an upper substrate surface to define an exposed portion of the substrate to be etched. The substrate, or batch of substrates, is then placed in the reactor chamber. Etching gases are introduced into the reactor chamber and a plasma is struck. During processing, the reactive species in the plasma etch the exposed portion of the metal, dielectric, or semiconductive material by contacting the exposed portion of the substrate.

At the molecular level, the etch process is a reaction between the reactive species in the plasma and the exposed surface layers of the substrate. The species

include free radicals, ions and other particles. Although the reaction between the substrate and the free radicals is essentially chemical in nature, it is greatly enhanced with the ion bombardment which contributes to the etching and provides activation energy to the surface reaction. The reaction between the plasma and a substrate yields
5 etch byproducts, *i.e.*, small volatile molecules that desorb from the surface and subsequently are diffused into the reactor chamber. Most of the volatile byproducts are then pumped out of the reactor chamber.

Etching of a single layer of material generally comprises two primary steps: a main etch process and an overetch process. The main etch process removes the bulk of
10 the material from the exposed substrate surface to form the desired feature. The overetch process is needed in order to remove residual material from the substrate while avoiding undercutting (*i.e.*, isotropic etching) and excessive loss of selectivity between interfacing layers, such as a polysilicon/oxide interface. The chemistry and process parameters for each step are selected to achieve anisotropy, constant, and
15 preferably high, etching rates, uniformity, high selectivity, and reproducibility.

Successful etching requires a controlled process to ensure uniform etching at a controlled and constant rate with respect to each individual substrate as well as from one substrate to the next. Uncontrolled changes in etching rates can lead to changes in device geometries and dimensions. Preferably, an etch rate change for a particular
20 substrate and from one substrate to the next is less than about 10%.

Process stability is affected by various methods and techniques commonly used in the industry. For example, a substantial change in the etch rate is observed after a cleaning process. Cleaning processes are periodically necessary to remove deposits of byproducts which form on the internal chamber surfaces during processing. Most
25 volatile byproducts formed during etching are pumped out of the chamber. However, the byproducts can often react with various gas components used in silicon etching with a halogen plasma, such as oxygen additives, to form less volatile byproducts. In other cases, the byproducts themselves can be less volatile, depending on the material being etched and the etch chemistry. These less volatile species can deposit onto the chamber
30 walls and other exposed surfaces enclosed within the chamber. Over time, the deposits can delaminate and flake off of the chamber surfaces creating a major source of particulate contamination. The particulate often become lodged in the mask or on the substrate surface and produce defective devices. As the size of the etched features

become smaller, the effects of particulate become more pronounced.

Thus, in order to control the contamination buildup, the chamber surfaces are cleaned periodically. One method of cleaning a chamber, known as dry cleaning, involves placing a dummy substrate in the chamber and then igniting a plasma. The plasma chemistry is selected to react both chemically and physically with the deposits on the chamber, thereby causing the deposits to form byproducts which can be pumped out of the chamber. However, a problem associated with chamber cleaning, is that the etching rates of the subsequent processes are adversely affected. In typical silicon plasma etching, etch rate drops in excess of 33% have been experienced following a chamber cleaning run. Changes in the etch rate are undesirable because of the resulting loss of process reproducibility, or repeatability. Because etching processes are often timed according to pre-programmed recipes, the fluctuation in etch rates results in over-etched or under-etched substrates. Consequently, repeatability between substrates is lost.

In order to minimize the detrimental effects of etch rate variation after a cleaning process, current practice employs a seasoning cycle during which the chamber is conditioned following a cleaning run. Seasoning refers to the operation of the chamber to allow deposition of a film on the chamber surfaces. During a recovery period a seasoning coating is allowed to form on the chamber surfaces by striking a plasma in the chamber and depositing a film on the internal exposed surfaces in the chamber. The chamber seasoning is continued until the pre-clean etch rate is completely recovered. The recovery period is time consuming and non-productive because no substrates are processed. Thus, the throughput of the system is significantly reduced.

Another problem related to process stability occurs when high concentrations of corrosive chemicals are used. For example, a problem exists in cases where fluorine is the major etchant, such as in planarization and recess-etching. Fluorine is a highly corrosive etchant that attacks the chamber surfaces causing a change in the topography of the surface and/or resulting in deposition of a byproduct, such as AlF_x in the case of an Al_2O_3 chamber surface. Unlike deposition of SiO_x which can be removed by non-intrusive cleaning methods such as plasma cleaning, as described above, the effects of fluorine etching must be treated by opening the chamber and refinishing the chamber surfaces. Thus, throughput is substantially affected. Additionally, following the

cleaning process, fluctuations in the etch rate are observed which inhibit etch rate stability.

Sensitivity to chamber conditions was also observed by the inventors in situations other than post-cleaning. For example, when the same chamber is used to run different applications involving alternating chemistry, varying etch rates are observed. Thus, processes alternating between fluorine-based chemistry and non-fluorine-based chemistry experience volatility in etch rates between each cycle. As an example, a chamber may first be used to process a number of substrates during a fluorine-based process such as a hardmask open or recess etching. Subsequently, the chamber may be used to run a second process not involving the use of fluorine, such as capacitor etching. For reasons heretofore not understood, the etch rates under such conditions experience fluctuation leading to loss of process repeatability.

Therefore, there is a need for reducing the process sensitivity of a processing chamber to the chamber surface conditions.

SUMMARY OF THE INVENTION

The present invention generally provides a method and apparatus of minimizing etch process sensitivity to chamber conditions. The etch process sensitivity can be controlled by various methods including changing the etch chemistry, process parameters and/or changing the chamber materials.

In one aspect of the invention, a composition of one or more etchants is selected to optimize the etch performance and reduce deposition on chamber surfaces. The one or more etchants are selected to minimize buildup on the chamber surfaces, thereby controlling the chamber surface condition to minimize changes in etch rates due to differing recombination rates of free radicals with different surface conditions, thereby allowing etch repeatability. In one embodiment, at least one etchant is introduced into the chamber and a plasma is struck to form at least a first free radical density from the first etchant. The etchant is selected to minimize the rate at which the free radical density reacts to produce deposition on the internal surfaces of the chamber and wherein the recombination rate of the free radical density on the deposition formed on the internal surfaces is substantially different than the recombination rate of the free radical density on the internal surface. In another embodiment, chlorine and bromine are introduced into a chamber at a first and a second flow rate, respectively; wherein

the first flow rate is higher than the second flow rate. Preferably, the ratio of chlorine to bromine is greater than about 3:1.

In another aspect of the invention, a first and second etchant are flowed into an etch chamber wherein the first etchant is adapted to clean the internal surfaces of the chamber and prevent the deposition of byproducts thereon while simultaneously etching a substrate. A substantially constant chamber surface profile is maintained in order to maintain a substantially constant recombination rate of plasma constituents with the chamber surfaces. Preferably, the first etchant is a fluorine-containing fluid and the second etchant is preferably a halogen, such as chlorine or bromine.

In yet another aspect of the invention an etch chemistry is selected to minimize corrosion of a chamber surface. A first etchant having corrosive properties is diluted with a second etchant wherein the second etchant is preferably non-corrosive or less corrosive than the first etchant. The first etchant is a fluid exhibiting corrosive effects when brought into contact with an internal chamber surface. The second etchant is combined with the first etchant to reduce the corrosive effects of the first etchant on chamber surfaces. The resulting mixture preferably exhibits reduced corrosive effects on chamber surfaces and allows successive processing of multiple substrates under substantially similar chamber surface conditions. In one embodiment, the first etchant is a fluorine-containing fluid such as CF_4 , SF_6 or NF_3 . The second etchant is preferably a chlorine-containing fluid such as Cl_2 .

In yet another aspect of the invention, a chamber pressure is controlled to reduce the sensitivity of an etch process to the chamber surface conditions. The chamber pressure is controlled during an overetch process to increase the ratio of ions to free radicals, thereby increasing ion-assisted etching and reducing pure free radical etching. In one embodiment, the pressure during an overetch of a polysilicon layer is preferably less than about 30 mTorr. The etch process is preferably bromine-based or chlorine-based.

In yet another aspect of the invention, the chamber pressure and the concentration of oxygen in the chamber is controlled to reduce the sensitivity of an etch process to the chamber surface conditions. The chamber pressure is controlled during an overetch process to increase the ratio of ions to free radicals, thereby increasing ion-assisted etching and reducing free radical etching. The oxygen concentration is controlled to reduce oxidation of a substrate surface being etched. In one embodiment,

the pressure during an overetch of a polysilicon layer is preferably less than about 10 mTorr and the oxygen concentration is less than about 25% of the total flow volume of gases into the chamber. The etch process is preferably bromine-based or chlorine-based.

5 In yet another aspect of the invention, the chamber pressure, the concentration of oxygen in the chamber, and the source power is controlled to reduce the sensitivity of an etch process to the chamber surface conditions. The chamber pressure is controlled to increase the ratio of ions to free radicals, thereby increasing ion-assisted etching and reducing free radical etching. The oxygen concentration is controlled to
10 reduce oxidation of a substrate surface being etched. The source power delivered to an inductive coil is adjusted to control substrate surface oxidation and etching. In one embodiment, the pressure during an overetch process of a polysilicon layer is preferably less than about 10 mTorr, the oxygen concentration is less than about 25% of the total flow volume of gases into the chamber and the source power is between
15 about 100-500W. Preferably, the oxygen concentration and the source power are inversely adjusted, such that when the oxygen concentration is increased, the source power is decreased and vice versa. The etch process is preferably bromine-based or chlorine-based.

In still another aspect of the invention, the chamber materials are selected to
20 reduce the sensitivity of an etch process to the chamber surface conditions. The materials are selected according to the recombination rate of free radicals therewith. Preferably, the recombination rate of the free radicals with the selected materials is substantially equal to the recombination rate of the free radicals with the byproducts formed during the etch process and deposited on the chamber surfaces. Thus, the etch
25 rate is substantially constant over time even where deposits are formed on the chamber surfaces. In one embodiment, the chamber components exposed to the processing environment of a silicon etch chamber, such as the chamber body and the chamber dome/lid, substantially comprise quartz. The chamber components may be constructed of quartz or, alternatively, may be lined with quartz.

30 In still another aspect of the invention, the chamber temperature is controlled to reduce the sensitivity of an etch process to the chamber surface conditions. The temperature is preferably sufficiently high to reduce deposition on exposed chamber surfaces. In one embodiment, a polysilicon layer is etched at a chamber temperature of

at least about 200°C.

BRIEF DESCRIPTION OF THE DRAWINGS

So that the manner in which the above recited features, advantages and objects
5 of the present invention are attained and can be understood in detail, a more particular
description of the invention, briefly summarized above, may be had by reference to the
embodiments thereof which are illustrated in the appended drawings.

It is to be noted, however, that the appended drawings illustrate only typical
embodiments of this invention and are therefore not to be considered limiting of its
10 scope, for the invention may admit to other equally effective embodiments.

Figure 1 is a schematic view of an etching chamber.

Figure 2 is a graphical representation of the deposition rates of etchants as a
function of oxygen.

15 Figure 3 is a graphical representation of an etch rate drop and free radical
density drop as a function of pressure for HBr-based etching.

Figure 4 is a graphical representation of an etch rate drop and free radical
density drop as a function of pressure for Cl₂-based etching.

20 Figure 5 is a graphical representation of an etch rate drop as a function of
oxygen for HBr-based etching.

Figure 6 is a graphical representation of an etch rate as a function of oxygen for
HBr-based etching.

Figure 7 is a graphical representation of an etch rate drop as a function of
oxygen for Cl₂-based etching.

25 Figure 8 is a graphical representation of an etch rate as a function of oxygen for
Cl₂-based etching.

Figure 9 shows graphical representations of etch rates as a function of oxygen at
varies source power levels for Cl₂-based etching.

30 Figure 10 is a graphical representation of an etch rate drop as a function of
source power.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The present invention generally relates to methods of minimizing etch process

sensitivity to chamber conditions. The etch process sensitivity can be controlled by various methods including changing the etch chemistry and/or process parameters and changing the chamber materials.

For clarity and ease of description, the following description refers primarily to a silicon etch system and the processes performed therein, including polysilicon etching. However, the invention is equally applicable to other types of processes that utilize chemicals to deposit or etch materials on or from a substrate and which are responsive to changing surface conditions. For example, chemical vapor deposition (CVD) processes may use the present invention to advantage.

One chamber 10 which may be used to advantage with the present invention is schematically illustrated in Figure 1. The chamber 10 includes a body 12 preferably made of a metal, such as anodized aluminum, and a dome ceiling 13 preferably made of a dielectric, such as ceramic. The chamber 10 defines a plasma zone 14 having a support member 16 disposed therein. The support member 16 may be a mechanical or electrostatic chuck with grooves in which a coolant gas, such as helium, is held to control the temperature of the substrate 20 disposed on the support member 16 during processing. At least a portion of the support member 16 is electrically conductive and serves as a process cathode. The support member 16 cooperates with the electrically grounded body 12, which serves as an anode, to form process electrodes in the plasma zone 14.

Process gas is introduced into the chamber 10 through a gas distributor 22 peripherally disposed about the support member 16. A plasma is formed from the process gas using a plasma generator that couples an electromagnetic field into the plasma zone 14. The plasma generator can comprise an inductor coil 26 disposed adjacent to the dome ceiling 13 of the process chamber 10. Preferably, the inductor coil 26 is wrapped around the dome ceiling 13 and is capable of forming an inductive electric field in the chamber when powered by a coil power supply 27.

In addition to the inductor coil 26, the support member 16 is powered by an electrode power supply 28 to generate a capacitive electric field in the process chamber 10. Typically an RF voltage is applied to the support member 16 while the body 12 is electrically grounded. The capacitive electric field is perpendicular to the plane of the support member 16, and accelerates inductively formed plasma species toward the substrate 20 to provide more vertically oriented anisotropic etching of the substrate 20.

Spent process gas and etchant byproducts are exhausted from the process chamber 10 through an exhaust system 30. A throttle valve 32 is provided in an exhaust port 34 for controlling the pressure in the chamber 10. Also, an optical endpoint measurement technique may be used to determine completion of the etching process for a specific layer by measuring the change in light emission of a particular wavelength corresponding to a detectable gaseous species. A sudden decrease or increase in the amount of the detectable species indicates completion of etching of one layer and the start of etching of another layer.

The processes described below were carried out in a chamber such as the one described above with reference to Figure 1. However, the particular processing chamber used is not limiting of the present invention. Other chambers may be used to advantage.

As described above, process stability is desired during etching in order to ensure repeatability of etch results. Further, it was noted above that various common procedures used in etch chambers, such as chamber cleaning, resulted in unacceptable levels of etch rate changes. The inventors have discovered that process stability is dependent upon passive chamber conditions such as the chamber wall surface condition. Frequently, these chamber conditions are altered without regard for, or awareness of, the resulting impact on the subsequent etching processes.

The inventors postulate that at least one cause of the fluctuation in etch rates is attributed to the different recombination rates of reactive free radicals on different chamber surfaces. For example, in the case of silicon etching, a byproduct identified as a SiO_x (where $x = 1-2$) compound is deposited on the internal chamber surfaces. After a cleaning cycle, the byproduct is removed and the underlying chamber surfaces are revealed. Where the recombination rates of radicals with the byproducts is lower than the recombination rates of radicals with typical chamber materials, such as ceramic and anodized aluminum, the free radical density in the plasma increases as the chamber surfaces are covered with the byproducts. Thus, when the chamber surfaces are free of deposition, such as immediately following a cleaning cycle, the free radicals recombine with the chamber surfaces at a first rate. Over a period of time, a layer of byproducts is formed on the chamber surfaces resulting in a second recombination rate of the free radicals with the chamber surfaces wherein the second rate is less than the first rate. Once the thickness of byproduct deposition exceeds an acceptable level, the chamber is

cleaned to again expose the underlying chamber surfaces, thereby resulting in the first recombination rate of free radicals for subsequent processes. Using optical actinometry, the inventors observed a 50%-70% decrease in the free radical density in Cl-based and Br-based etching following a cleaning run. The varying recombination rates result in fluctuating radical densities in the plasma available for etching a substrate and the etch rate is varied over time as a function of the chamber surface composition and condition.

In general, for polysilicon etch processes, the etch rate is increased as deposits such as SiO_x (where $x = 1-2$) are formed on the chamber surfaces. However, any change in the etch rate which significantly impairs the consistency and repeatability of an etch process is considered detrimental. The effects of varying free radical densities are particularly detrimental in cases where the process involves reactions dominated by free radical concentrations such as the overetch process in an etch process. As noted above, the overetch process is a process where residual material from the substrate is selectively removed to form the features of a substrate under highly controlled conditions.

In one embodiment of the invention, the etch chemistry is adjusted to minimize deposition of byproducts on the chamber surfaces when the recombination rate of radicals with the material deposited on the chamber surfaces is substantially different from the recombination rate with the chamber surface material. A primary etchant is selected according to the etchant's potential to deposit byproducts on chamber surfaces. The primary etchant is defined as the etchant having the highest volumetric flow relative to each of the individual constituents of the chemical mixture flowed into the chamber and may be the only etchant used in some cases. Preferably, the primary etchant is selected to minimize the formation of byproducts that are deposited on chamber surfaces.

One example of the present invention is illustrated with reference to Tables I and II. Table I shows a conventional "baseline" process recipe for a main etch process and an overetch process of silicon. The main etch process consists of halogens, such as Cl and Br mixtures, with proportionately higher Br content. The overetch process consists of Br-based chemistry and is performed at higher pressures relative to the main etch process. The use of Br-containing plasmas for the main etch process as well as the overetch process provides high selectivity anisotropic poly etch. Compared to Cl, Br is

found to increase the poly:oxide selectivity and allow greater control of the etching profile. The process parameters given in Table I are typical for etching a polysilicon layer on a substrate.

TABLE I

Parameter	Main Etch	Overetch
Composition:	Cl ₂ , HBr (1Cl:2Br), O ₂ (< 5% by volume)	HBr O ₂ (< 3% by volume)
Flow rate:	200 sccm	155-160 sccm
Coil Power:	500W	500-1000W
Bias Power:	80W	100-150W
Pressure:	4mTorr	50mTorr
Temperature:	50°C	50°C

The inventors have determined that, contrary to the trend and common expectations, higher proportions of Cl minimize the detrimental effects of changing chamber surface conditions, such as the rate variation in overetch where the etching is dominated by free radicals. Figure 2 is a comparative graph showing a deposition curve for HBr etching and a deposition curve for Cl₂ etching of 3000Å of polysilicon.

The curves plot the deposition thickness of SiO_x (where x = 1-2) on a chamber surface in changing O₂ concentrations. The parameters for both curves were substantially the same and are as follows. Cl₂-based: 50°C, 100 sccm (Cl₂ and O₂), 3mTorr, 400W (coil), 60W (bias). HBr-based: 50°C, 100 sccm (HBr and O₂), 6mTorr, 400W (coil), 60W (bias).

Figure 2 indicates that the use of Br-based chemicals, such as HBr, result in higher levels of deposits in the chamber as compared to deposits resulting from Cl-based chemicals, such as Cl₂. This result is attributed to the fact that during poly etching, SiCl and SiBr compounds are formed. SiCl is volatile and is pumped out of the chamber. SiBr, however, is less volatile and reacts with the O₂ to produce SiO_x compounds that are deposited onto the chamber surfaces.

As described above, the density of free radicals in the plasma is directly related to the chamber surface condition. Because the recombination rates of radicals with SiO_x is lower than the recombination rates of radicals with typical chamber materials,

such as ceramic and anodized aluminum, the free radical density in the plasma increases as the chamber surfaces are covered with SiO_x. Thus, as the chamber surface condition changes over time, the etch rate changes accordingly. Specifically, because the free radicals contribute to the etching of the polysilicon, the etch increases with increasing deposits on the chamber surfaces. Such variation in the etch rate is undesirable because etch repeatability is compromised. The free radical density is particularly important during processes which are chemical in nature, such as the overetch process, and conventionally rely on high concentrations of radicals to maintain a desired etch rate. A change in the density of free radicals results in varying overetch rates, and consequently a loss of repeatability. Thus, successful etching requires the etch rate to be controlled and to be substantially constant over time.

The inventors have determined that adjusting the etch chemistry to consist of higher proportions of Cl relative to Br can reduce deposition of SiO_x compounds on chamber surfaces during etching. Minimizing the deposit buildup on the chamber surfaces results in a substantially constant density of free radicals in the etching plasma and, therefore, a substantially constant etch rate.

Table II shows a process recipe according to the present invention.

TABLE II

Parameter	Main Etch	Overetch
Composition:	Cl ₂ , HBr (4Cl:1Br), O ₂ (< 5% by volume)	HBr, O ₂ (< 3% by volume)
Flow rate:	100-200 sccm	100-200 sccm
Coil Power:	300-500W	300-500W
Bias Power:	80W	100W
Pressure:	4mTorr	50mTorr
Temperature:	10-30°C	10-30°C

Comparison of Table I and Table II reveals a marked departure from conventional recipes. The process recipe of the invention preferably comprises a Cl:Br ratio between about 3:1 and 5:1. Most preferably, the ratio of Cl:Br is about 4:1, as shown in Table II.

The process according to Table I leads to substantial deposition on the internal

chamber surfaces over a short period time. The deposition rate of SiO_x compounds was measured as 50-100 Å/min. In contrast, the process recipe of Table II resulted in a deposition rate of only 10-30 Å/min. The reduced deposition rate is attributed to the higher proportions of Cl relative to Br.

5 While Br is the primary etchant used during the overetch process in Table II, the resulting byproduct deposition on chamber surfaces is negligible. The overetch process serves only to remove residual material from the device features substantially formed during the main etch process. Thus, the undesirable chamber surface deposition is formed primarily during the main etch process while the overetch process contributes
10 only negligible amounts of deposits on the chamber surfaces.

However, Cl and Br-based etching is merely one example of processes which may be used to advantage according to the invention. The invention is applicable in any plasma process resulting in deposition of byproducts on chamber surfaces which effects a change in the recombination rate of free radicals. Such processes include
15 plasma enhanced chemical vapor deposition, for example. Further, although the foregoing description with reference to Tables I and II illustrates the impact of a silicon main etch on an overetch, the invention contemplates application to any step or steps which alter the chamber surface conditions.

In addition to the etch chemistry, the process parameters, such as pressure, also
20 impact the etch rate. Referring again to Table I, the processing pressure in the baseline overetch recipe is 50mTorr. Conventionally, process pressures in the overetch process are 50 mTorr or higher because higher pressures afford higher poly:oxide selectivity. At pressures of 50 mTorr and higher, etching is typically more chemical in nature because the radical-to-ion ratio is higher as compared to lower pressures. As the
25 pressure decreases, the radical-to-ion ratio also decreases resulting in an ion dominant, or ion-assisted, etching. Ion-assisted etching refers to etching done by free radicals wherein ion bombardment contributes to the activation energy of the surface reaction. Ion-assisted etching reduces the sensitivity of the etch process to the free radical density, thereby reducing the etch sensitivity to free radical density fluctuations with
30 changing chamber wall conditions.

Figure 3 shows the overetch rate drop and free radical density drop as a function of pressure for Br-based etching. The etch rate/density drop is calculated as the percentage decrease in the overetch rate/density after a dry clean process as compared

to the overetch rate/density prior to the dry clean process wherein the chamber surfaces had accumulated buildup. The process recipe for the overetch of Figure 3 was as follows: 150 sccm HBr and 10 HeO₂ sccm, 50°C, 1000W (source power), 100W (bias power). At a pressure of about 4 mTorr the corresponding etch rate drop is only about 1% and the Br density drop is about 50%. As the pressure is increased, the etch rate drop and the density drop also increase. At a pressure of about 50 mTorr, the etch rate drop is about 33% and the free radical density drop is about 58%. Figure 4 shows an etch rate drop and free radical density as a function of pressure for Cl₂-based etching. Again, at increasing pressures the etch rate drop and the density drop both increase with increasing pressure as the etching becomes more dependent on free radicals.

The inventors postulate the substantial etch rate drop at higher pressures is due to the recombination rates of radicals with the internal chamber surfaces. After a cleaning process, the recombination rate of the radicals with the chamber surfaces is higher than during pre-cleaning processes wherein the underlying chamber surfaces are covered by deposits. As a result of the cleaning, the underlying chamber surfaces are exposed. Because the recombination rate of free radicals with the underlying chamber surfaces is higher than the recombination rate with the chamber deposits, the density of available radicals in the plasma is reduced resulting in an etch rate drop in process steps relying on high concentrations of radicals, such as the overetch process. However, at lower pressures the etching becomes ion dominated and the chemical etching due to free radicals is reduced. Decreasing the pressure decreases the free radical to ion ratio, thereby increasing the ion etching and decreasing the free radical etching. As a result, the process becomes less dependent on the free radical density.

Thus, the effects of cleaning a chamber on the overetch process can be mitigated according to the low pressure processing of the present invention. In a preferred embodiment, the overetch process recipe for polysilicon comprises a pressure of less than about 30 mTorr and most preferably comprises a pressure of about 10mTorr. The pressure may be optimized for a particular etch chemistry and process parameters. Preferably, the etch rate change after a cleaning process is less than about 10%. Thus, contrary to conventional practice, the invention provides a low pressure method of overetching to minimize the effects of the chamber wall condition. This solution is counter-intuitive because the selectivity is sacrificed as the pressure is lowered. However, selectivity at the lower pressures of the present invention may be

increased by controlling other process parameters. For example, the bias power may be decreased to lessen the rate at which ions etch the substrate. Further, the oxygen flow rate may be increased to increase oxidation of the substrate surface, thereby decreasing the etch rate.

5 In another embodiment, the results of low pressure overetching are further improved by optimizing the concentrations of oxygen which is an etch chemistry additive used to achieve higher selectivity. Figures 5 and 6 show the overetch rate drop and the overetch rate, respectively, for HBr-based etching. Figures 7 and 8 show the overetch rate drop and the overetch rate, respectively, for Cl₂-based etching. Both HBr
10 and Cl₂-based etching were detrimentally affected by increasing concentrations of oxygen due to surface oxidation of the substrate.

After a dry clean process, the concentrations of free radical etchants, *e.g.*, Cl and Br, are decreased resulting in a proportionally higher concentration of oxygen. The oxygen oxidizes the substrate surface being etched, thereby inhibiting reactions
15 between the etchants and the substrate surface. Consequently, the etch rates are decreased. While some oxygen is desirable to achieve greater selectivity, the oxygen concentration must be controlled to avoid a detrimental impact on the etch rate due to excessive oxidation of the substrate surface. As shown in Figures 5 and 7, the preferred oxygen concentration is also dependent on the etchant chemistry used. For HBr-based
20 etching, shown in Figure 6, the oxygen concentration is preferably between about 0% and 5%. For Cl₂-based etching, shown in Figure 8, the oxygen concentration is preferably between about 0% and 15%.

However, the optimal oxygen concentrations also vary according to other process parameters and must be adjusted accordingly. For example, the preferred
25 oxygen concentration is dependent on the source power, *i.e.*, the inductive coil power supplied to the inductive coil such as the coil 26 shown in Figure 1. Figure 9 shows two graphs for Cl₂-based etching where the Cl₂ flow rate is about 50sccm, the bias power to the support member is about 40W, and the chamber pressure is at about 3mTorr. A first graph 50 plots the effects of oxygen concentration on the overetch rate at about 200W and a second graph 52 plots the effects of oxygen concentration on the
30 overetch rate at about 400W. Graph 50 indicates a peak etch rate at an oxygen concentration of about 10% and a source power of about 400W. Graph 52 indicates a peak etch rate at an oxygen concentration of about 15% about and a source power of

about 200W.

While both graphs exhibit decreasing etch rates with increasing oxygen concentration after critical points 54 and 56, graph 52 sustains a higher etch rate at higher concentrations of oxygen. Thus, at about 25% oxygen, graph 50 exhibits an etch rate of about 800 Å/min while graph 52 exhibits an etch rate of about 2000 Å/min. Thus, according to the invention, the oxygen concentrations and source power levels are adjusted to optimize the etch rate. Where higher oxygen levels are desirable, the source power levels are reduced to accommodate optimal etching rates. Conversely, where the lower oxygen levels are preferred, higher levels of source power may be used to achieve optimal etching rates. Although not shown in Figure 9, HBr and other etchants exhibit similar responsiveness to changes in source power levels in the presence of oxygen.

However, at an upper limit the source power causes an etch rate drop, as shown by the downward sloping portions of graphs 50 and 52 after the critical points 54 and 56. The etch rate drop is a result of accelerated surface oxidation at higher source power levels. Figure 10 represents the etch rate drop for an HBr-based etch process having an oxygen concentration of about 4%. At about 600W of source power, the etch rate drop increases abruptly as a result of excessive surface oxidation on the substrate. Thus, the optimal processing recipe is achieved by controlling the level of oxygen and source power.

Two embodiments of a low pressure overetch process in the presence of oxygen are shown in Table III. In one embodiment, Cl-based chemistry is used. In another embodiment, Br-based chemistry is used.

TABLE III

<u>Parameter</u>	<u>Cl-based overetch</u>	<u>Br-based overetch</u>
Composition:	Cl ₂ , O ₂ (< 15% by volume)	HBr, O ₂ (< 5% by volume)
Flow rate:	50 sccm	100 sccm
Coil Power:	100-200W	300-500W
Bias Power:	30-50W	50-100W
Pressure:	3-5mTorr	10-30mTorr
Temperature:	0-10°C	30-50°C

While Table III shows only Cl-based chemistry and HBr-based chemistry for silicon etching, the invention contemplates other processes which are conventionally done in high pressure regimes and are sensitive to surface conditions. The optimal parameters for a given etch chemistry are determined according to the relationship between pressure, oxygen concentration, and source power as disclosed herein.

Another embodiment of the invention comprises the use of cleaning agents during etching. The term "cleaning agent" refers to highly volatile gases often used to clean chamber surfaces periodically. The present invention utilizes such gases to advantage during etch sequences. Thus, the cleaning agents provide a dual function of etching while also maintaining a clean chamber surface.

One cleaning gas which may be used to advantage during the main etch process is fluorine. Table IV and Table V provide preferred process recipes of the invention.

TABLE IV

Parameter	Main Etch: Cl-based	
Composition:	Chlorine-containing etchant:	Cl ₂ ; and
	Fluorine-containing chemical:	CF ₄ (< 50% by volume),
	SF ₆ (< 20% by volume) or NF ₃ (<20% by volume)	
Flow rate:	200 sccm	
Coil Power:	300-500W	
Bias Power:	50-100W	
Pressure:	4mTorr	
Temperature:	30-50°C	

TABLE V

Parameter	Main Etch: HBr-based and/or HCl-based	
Composition:	Etchant:	HBr and/or HCl; and
	Fluorine-containing chemical:	SF ₆ (< 20% by volume),
	NF ₃ (<20% by volume), or CF ₄ and O ₂ (< 50% by volume, where CF ₄ :O ₂ = 4:1)	
Flow rate:	200 sccm	
Coil Power:	300-500W	

Bias Power: 50-100W
Pressure: 4mTorr
Temperature: 30-50°C

5 Table IV provides a preferred recipe using Cl-based etchant chemistry. Table V provides a preferred recipe using HCl and/or HBr-based etchant chemistry. Both recipes are main etch process recipes. The use of volatile cleaning agents according to the invention is well suited for main etch processes where the bulk of the material from device features is removed because of the high etch rate. The subsequent overetch
10 process, which requires greater selectivity, may be performed according to any conventional recipe or a recipe of the invention and disclosed herein.

In operation, the internal chamber surfaces are exposed to a cleaning agent, such as a fluorine-based fluid, which reacts with byproducts formed during the main etch process and deposits formed on the chamber surfaces while also etching the desired
15 areas of the substrate. The fluorine-based fluid and the byproducts form volatile compounds which are exhausted from the chamber. Thus, deposition on the chamber surfaces is minimized and the chamber surfaces maintain a substantially consistent composition. As a result of maintaining constant chamber surface conditions, the recombination of free radicals with deposits on the chamber surfaces is kept
20 substantially constant over a longer period of time than is possible with conventional etching methods. Because the chambers do not require periodic cleaning, the up-time between cleaning cycles is increased to allow for improved productivity of the etching system.

While the addition of some fluorine-containing gases to processes producing
25 substantial amounts of byproducts prevents the byproducts from depositing on chamber surfaces, a problem exists in cases where fluorine is the major etchant, such as in planarization and recess-etching. Fluorine is a highly corrosive etchant that attacks the chamber surfaces. Changes in the chamber surface can include the formation of a byproduct, due to corrosion, whereon free radicals exhibit a recombination rate
30 different from the recombination rate on clean chamber surfaces. Additionally, chamber surface changes include changes in the texture of the internal chamber surfaces which can increase the exposed surface area, thereby providing increased recombination sites for free radicals. Over time, the chamber surfaces must be

refinished, or in extreme cases, the chamber components, such as the body and dome, must be discarded and replaced. Otherwise, the etch result will change with the changing surface condition.

The inventors have discovered that the etch chemistry may be altered to reduce the corrosive effects of fluorine-based etching on chamber surfaces. In one embodiment, Cl is added to the etch chemistry. Table VI shows a preferred recipe of the present invention involving silicon etching.

TABLE VI

10	<u>Parameter</u>	<u>Main Etch for Silicon Etching</u>
	Composition:	Chlorine-containing etchant: Cl ₂ ; and Fluorine-containing chemical: CF ₄ , SF ₆ , NF ₃ (< 50% by volume)
15	Flow rate:	200 sccm
	Coil Power:	500-1000W
	Bias Power:	0-50W
	Pressure:	4-20mTorr
	<u>Temperature:</u>	<u>50°C</u>

20

In general, a ratio of Cl to F, Cl:F, is preferably between about 1:2 to 2:1 and most preferably about 1:1. The addition of Cl to F-based chemistry is found to reduce to the corrosive impact of fluorine on chamber surfaces. Thus, the chamber condition is maintained substantially constant over a longer period of time than is possible using conventional recipes. As a result, the recombination rates of free radicals on the chamber surfaces are kept substantially constant during the processing of a substrate as well as from one substrate to the next.

25

In another embodiment of the invention, the chamber wall temperature is adjusted to reduce the deposition of byproducts on the chamber surfaces. Currently, the chamber wall temperature is maintained at about 60-80°C. The inventors have discovered that the deposition of byproducts on chamber surfaces decreases with increasing temperature. Therefore, the temperature can be controlled to reduce the amount of byproduct deposits on the chamber surfaces during processing. Preferably, the chamber wall temperature is higher than about 200°C to minimize deposition

buildup on the chamber surfaces, thereby preventing changes in the surface profile during processing.

In another embodiment of the invention, the etch chamber materials are selected to reduce the sensitivity of the etch process to changing surface conditions. As described above, one cause of etch rate fluctuation after a chamber cleaning cycle is the free radical recombination rates of etchants on varying surfaces. Thus, while the free radicals may have a first recombination rate on chamber surfaces comprising a first material such as aluminum or ceramic, the free radicals may have a second recombination rate on the process byproducts which are deposited on the chamber surfaces during processing. Over time, the chamber surface materials are covered by the byproduct deposits, thereby altering the surface composition exposed to the etchants and resulting in etch rate changes.

Conventionally, the effects of changing free radical recombination rates are minimized or eliminated by a seasoning cycle wherein a film comprising the byproducts formed during etching is deposited on the chamber to cover the chamber surface material. However, the seasoning cycle is time consuming and reduces the system throughput because production must be stopped.

The method and apparatus of the invention involves the selection of chamber materials according to the free radical recombination rates therewith. Preferably, the free radical recombination rates with the chamber material is substantially equal to the recombination rates with the byproduct deposition material which forms on the chamber surfaces over time. Thus, a substantially constant etch rate is maintained during changing chamber surface conditions. Preferably, the selected materials comprise at least 80% of the internal surface of the chamber.

In one embodiment, a poly etch chamber comprises an internal surface made of one or more materials such as quartz. However, the selected material is dependent on the etch chemistry. In each case, the material is selected to exhibit a free radical recombination rate thereon substantially equal to the recombination rate of the deposition which forms on the chamber. Referring to Figure 1, the selected materials are preferably used to form the internal surfaces of the chamber body 12 and/or the chamber dome lid 13.

The foregoing embodiments were presented separately, in part, for clarity. However, the present invention contemplates the use of one or more of the

embodiments described above in combination to optimize etching rates, repeatability and selectivity.

While foregoing is directed to the preferred embodiment of the present invention, other and further embodiments of the invention may be devised without
5 departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

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CLAIMS:

- 1 1. A method of etching substrates in a chamber having an internal surface,
2 comprising:
3 (a) introducing at least a first etchant into the chamber; and
4 (b) striking a plasma in the chamber to cause disassociation of the first
5 etchant, wherein the first etchant is selected to minimize deposition of a material on the
6 internal surface and wherein a first recombination rate of the disassociated etchant on
7 the material is substantially different than a second recombination rate of the
8 disassociated etchant on the internal surface.
- 1 2. The method of claim 1, wherein a deposition rate at which the material is
2 formed on the internal surface is less than about 30 Å/min.
- 1 3. The method of claim 1, further comprising:
2 (c) cleaning the internal surface prior to (a).
- 1 4. The method of claim 1, wherein the first etchant comprises chlorine.
- 1 5. The method of claim 4, further comprising:
2 (c) etching one or more layers from a substrate comprising silicon.
- 1 6. A method of etching a substrate in a chamber having an internal surface,
2 comprising:
3 (a) flowing at least a first etchant and a second etchant into the chamber,
4 wherein a volumetric flow of the first etchant is greater than a volumetric flow of the
5 second etchant; and
6 (b) striking a plasma in the chamber to cause disassociation of the first
7 etchant and the second etchant, wherein the disassociated first etchant deposits
8 material on the internal surface at a first rate and the disassociated second etchant
9 deposits material on the internal surface at a second rate less than the first rate.
- 1 7. The method of claim 6, wherein the first etchant comprises chlorine.

- 1 8. The method of claim 6, wherein the second etchant comprises bromine.
- 1 9. The method of claim 6, wherein the first etchant comprises chlorine and the
2 second etchant comprises bromine.
- 1 10. The method of claim 6, further comprising:
2 (c) etching one or more layers from the substrate, wherein the one or more
3 layers comprise silicon.
- 1 11. The method of claim 6, further comprising cleaning the internal surface prior to
2 (a).
- 1 12. The method of claim 6, further comprising flowing oxygen into the chamber.
- 1 13. The method of claim 12, wherein the first etchant comprises chlorine and the
2 second etchant comprises bromine.
- 1 14. A method of etching substrates in a chamber having an internal surface and
2 minimizing the effects of the internal surface condition on an etch process, comprising:
3 (a) etching a first substrate in the chamber at a first pressure according to a
4 main etch recipe; and
5 (b) etching the first substrate in the chamber at a second pressure less than
6 about 30mTorr according to an overetch recipe .
- 1 15. The method of claim 14, wherein the substrate comprises a polysilicon layer
2 disposed thereon and the overetch recipe comprises flowing one or more of a bromine-
3 containing fluid, a chlorine-containing fluid or combination thereof into the chamber.
- 1 16. The method of claim 14, wherein (b) comprises flowing a chemical mixture into
2 the chamber comprising a bromine-containing fluid and an oxygen-containing fluid,
3 wherein the oxygen-containing fluid is about 10% or less of the volumetric flow of the
4 chemical mixture.

1 17. The method of claim 14, wherein (b) comprises flowing a chemical mixture into
2 the chamber comprising a chlorine-containing fluid and an oxygen-containing fluid,
3 wherein the oxygen-containing fluid is about 25% or less of the volumetric flow of the
4 chemical mixture.

1 18. A method of etching a substrate, comprising:
2 (a) positioning the substrate in a chamber having an internal surface;
3 (b) flowing a chemical mixture into the chamber;
4 (c) striking a plasma in the chamber from the chemical mixture to form one
5 or more plasma constituents, and
6 (d) depositing a film on the internal surface;
7 wherein a first recombination rate of the one or more plasma constituents with
8 the internal surface is substantially equal to a second recombination rate of the one or
9 more plasma constituents with the film.

1 19. The method of claim 18, wherein the substrate comprises polysilicon and
2 wherein the internal surface substantially comprises quartz.

1 20. The method of claim 19, wherein the internal surface comprises a liner disposed
2 on a chamber body.

1 21. A method of etching a substrate, comprising:
2 (a) inserting the substrate into a chamber;
3 (b) flowing a chemical mixture into a chamber, the chemical mixture
4 comprising:
5 (i) one or more of a bromine-containing fluid and a chlorine-
6 containing fluid; and
7 (ii) a fluorine-containing fluid;
8 wherein a volumetric flow of the one or more of the bromine-containing
9 fluid and the chlorine-containing fluid is at least 50% of the chemical mixture;
10 and
11 (c) striking a plasma.

1 22. The method of claim 21, wherein the fluorine-containing fluid comprises one or
2 more of SF₆, NF₃ and any combination thereof and wherein a volumetric flow of the
3 fluorine-containing fluid is less than about 20% of the chemical mixture.

1 23. The method of claim 21, wherein fluorine-containing fluid comprises CF₄ and
2 O₂ and wherein a volumetric flow of the fluorine-containing fluid is less than about
3 50% and a volumetric flow ratio of CF₄ to O₂ is about 4:1.

1 24. The method of claim 21, wherein the fluorine-containing fluid comprises one or
2 more of CF₄ and a volumetric flow of CF₄ is less than 50% of the chemical mixture.

1 25. The method of claim 21, wherein the bromine-containing fluid comprises
2 hydrogen bromide and the chlorine-containing fluid comprises hydrogen chloride.

1 26. The method of claim 21, further comprising:
2 (c) etching one or more layers from the substrate, wherein the one or more
3 layers comprise silicon.

1 27. An apparatus for etching a substrate, comprising:
2 (a) a process chamber including a chamber body having an internal surface,
3 ;
4 (b) one or more sources of one or more etchants coupled to the process
5 chamber; and
6 (c) at least one coil disposed adjacent the process chamber for striking a
7 plasma in the process chamber by disassociating the one or more etchants, wherein a
8 first reaction rate of the disassociated one or more etchants with the internal surface is
9 substantially the same as a second reaction rate of the disassociated one or more
10 etchants with a material formed on the internal surface during processing.

1 28. The apparatus of claim 27, wherein the substrate comprises polysilicon and
2 wherein the internal surface substantially comprises quartz.

- 1 29. The apparatus of claim 27, wherein the internal surface comprises a liner
- 2 disposed on the chamber body.

ABSTRACT OF THE DISCLOSURE

A method and apparatus for reducing the sensitivity of semiconductor processing to chamber conditions is provided. Process repeatability of common processes are affected by changing surface conditions which alter the recombination rates of processing chemicals to the chamber surfaces. In one aspect of the invention, a composition of one or more etchants is selected to optimize the etch performance and reduce deposition on chamber surfaces. The one or more etchants are selected to minimize buildup on the chamber surfaces, thereby controlling the chamber surface condition to minimize changes in etch rates due to differing recombination rates of free radicals with different surface conditions and achieve etch repeatability. In another embodiment, the etchant chemistry is adjusted to reduce the change to internal surface conditions after a cleaning cycle. In another embodiment, a process recipe is selected to reduce the sensitivity of the etch process to the chamber conditions. In another embodiment, chamber surface materials are selected to minimize the differences in recombination rates of free radicals on the surface materials and the byproduct depositions formed on the materials during processing.

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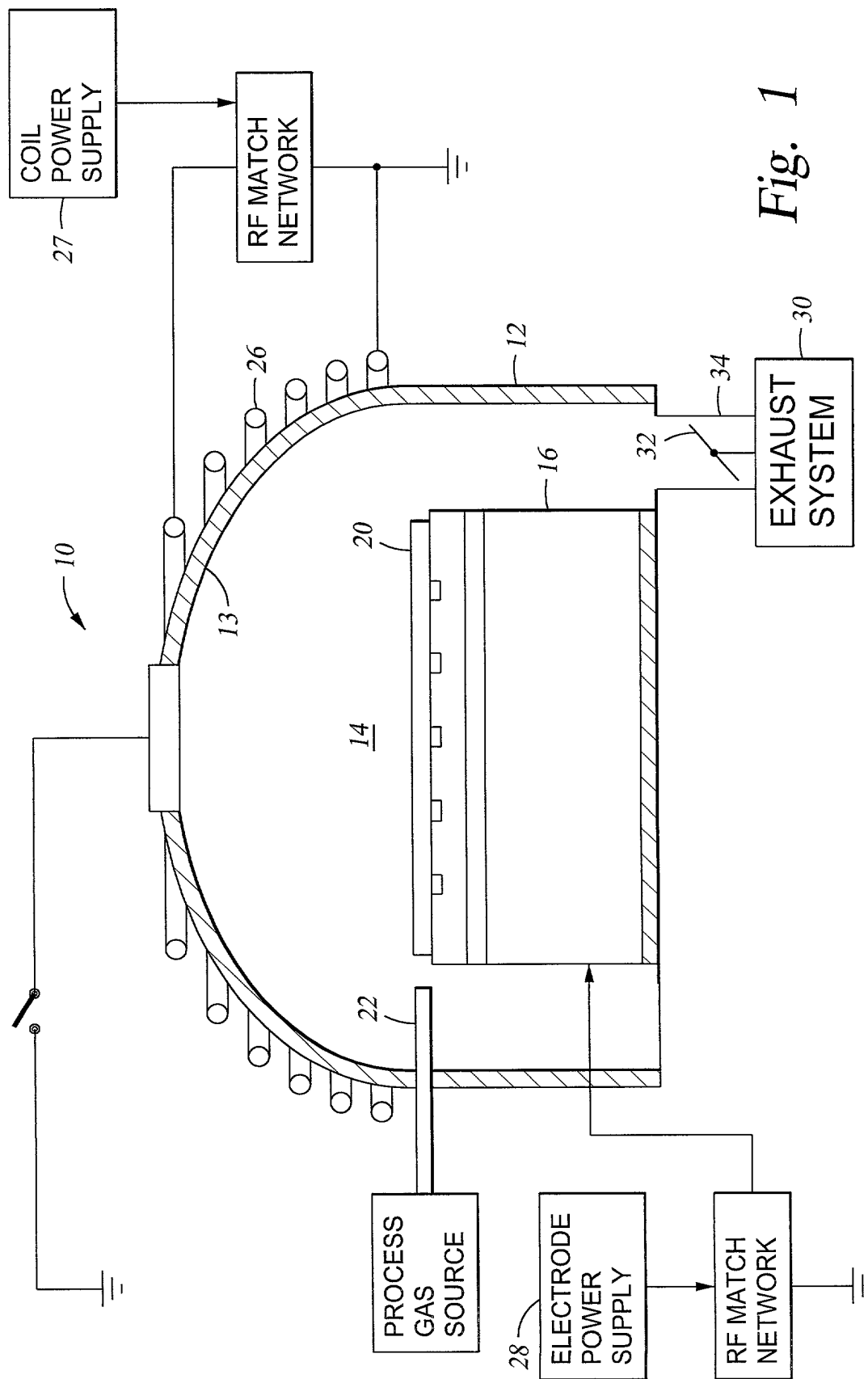
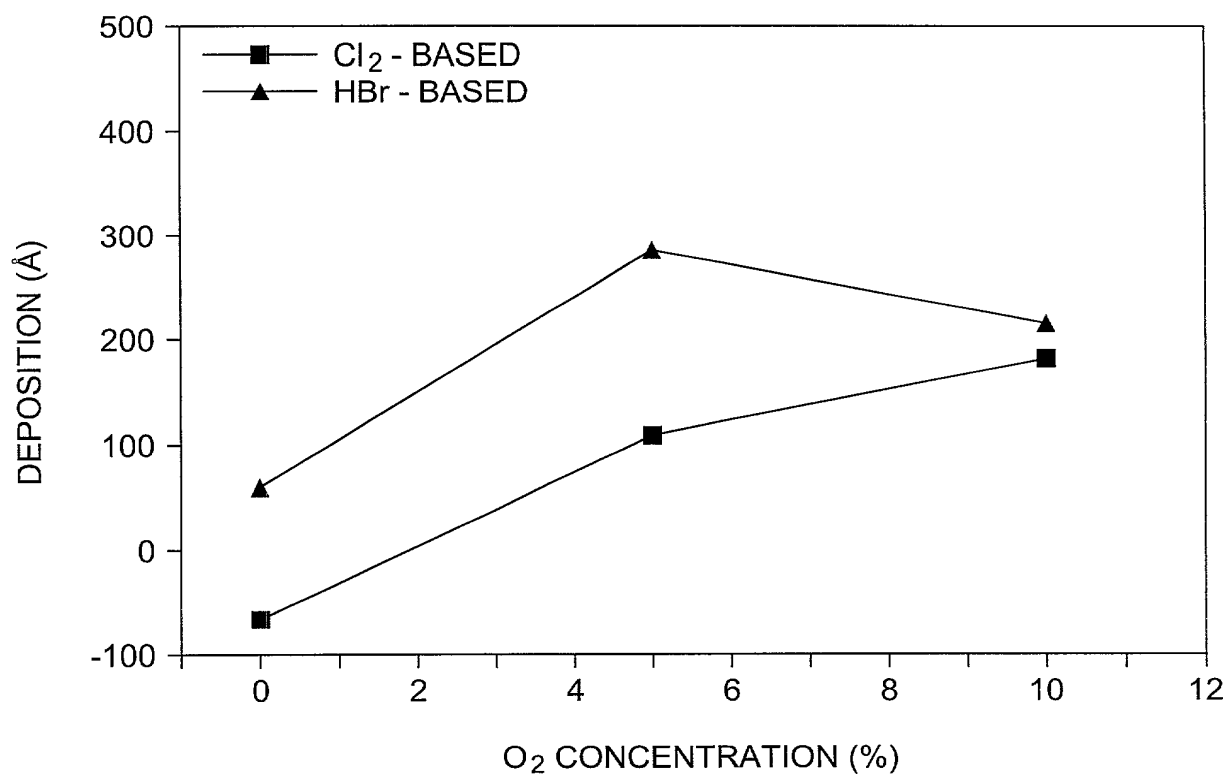
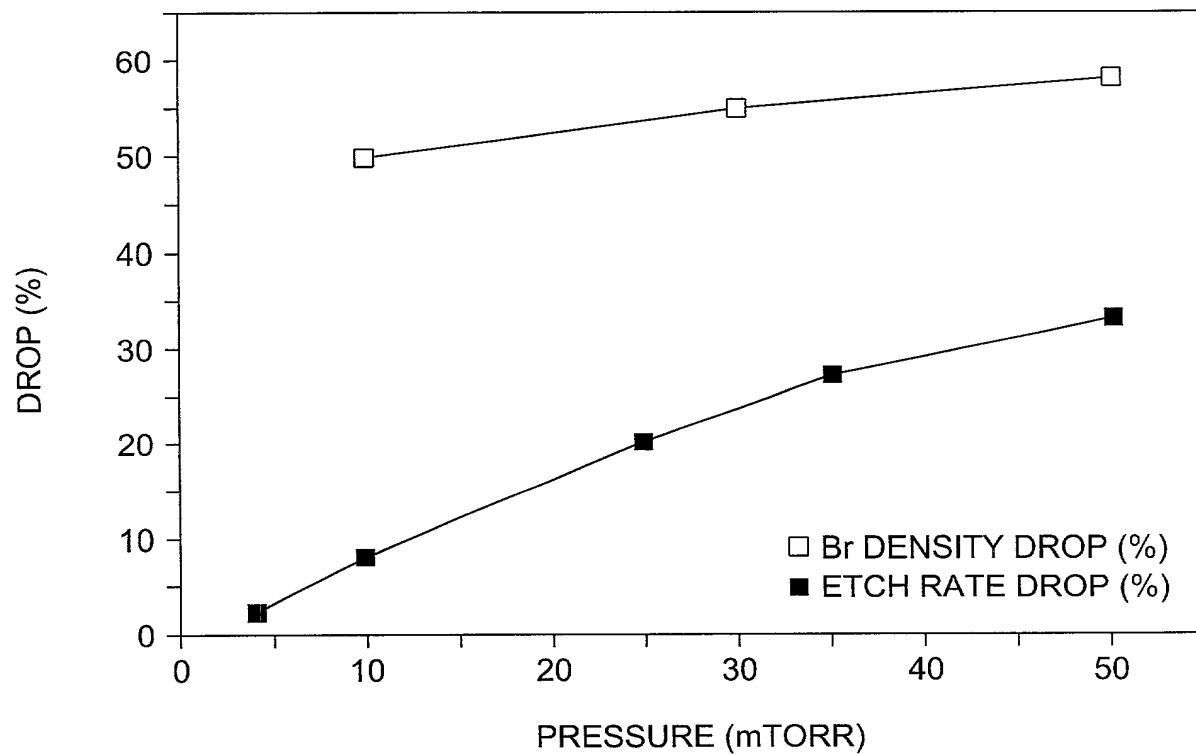
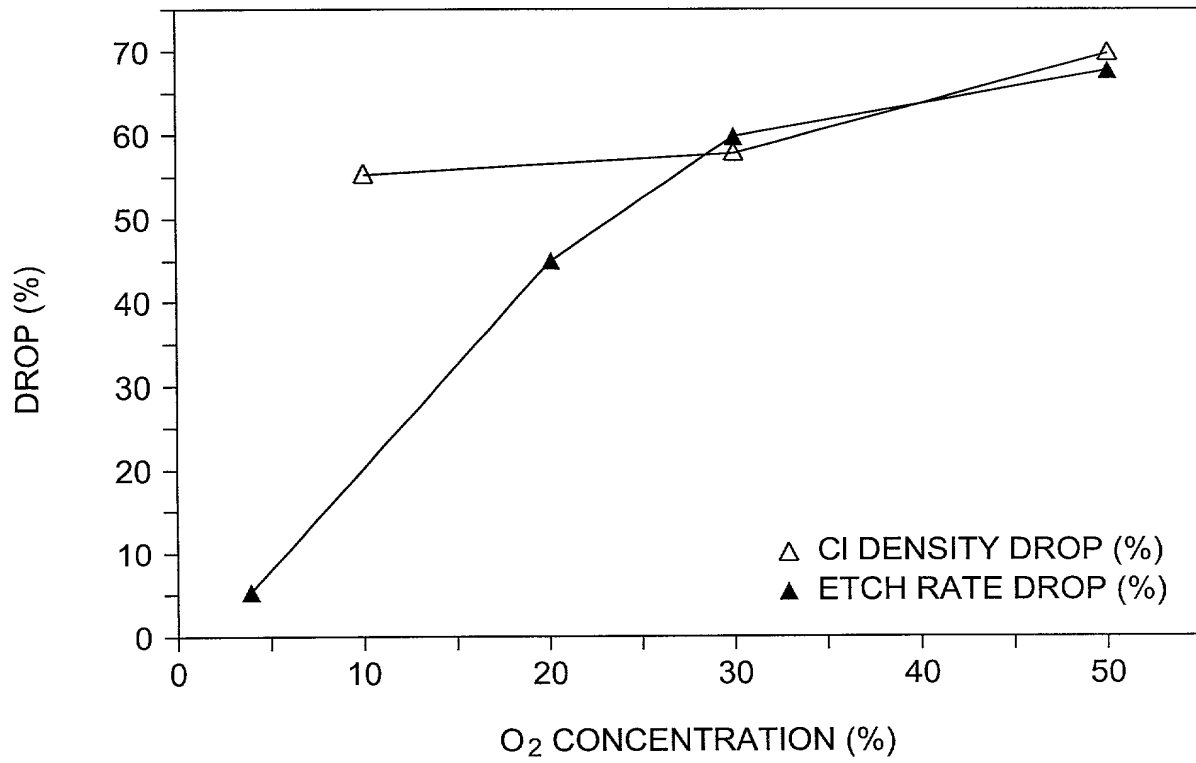
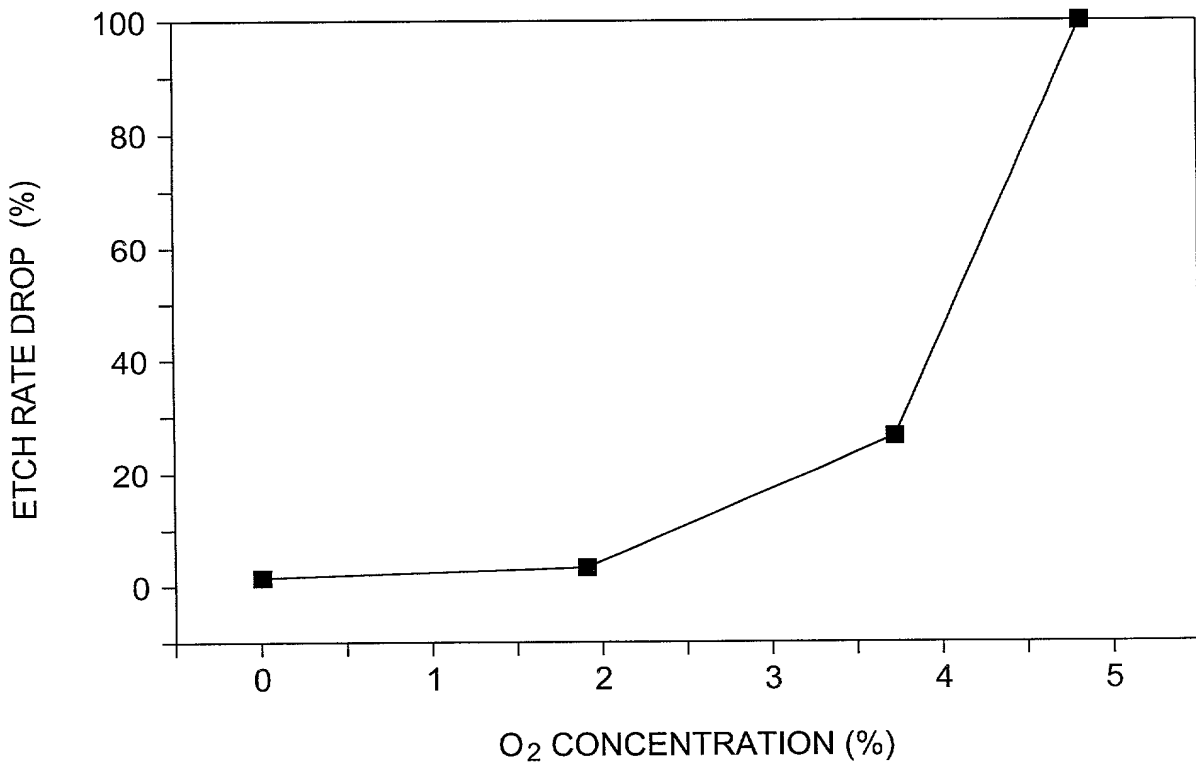
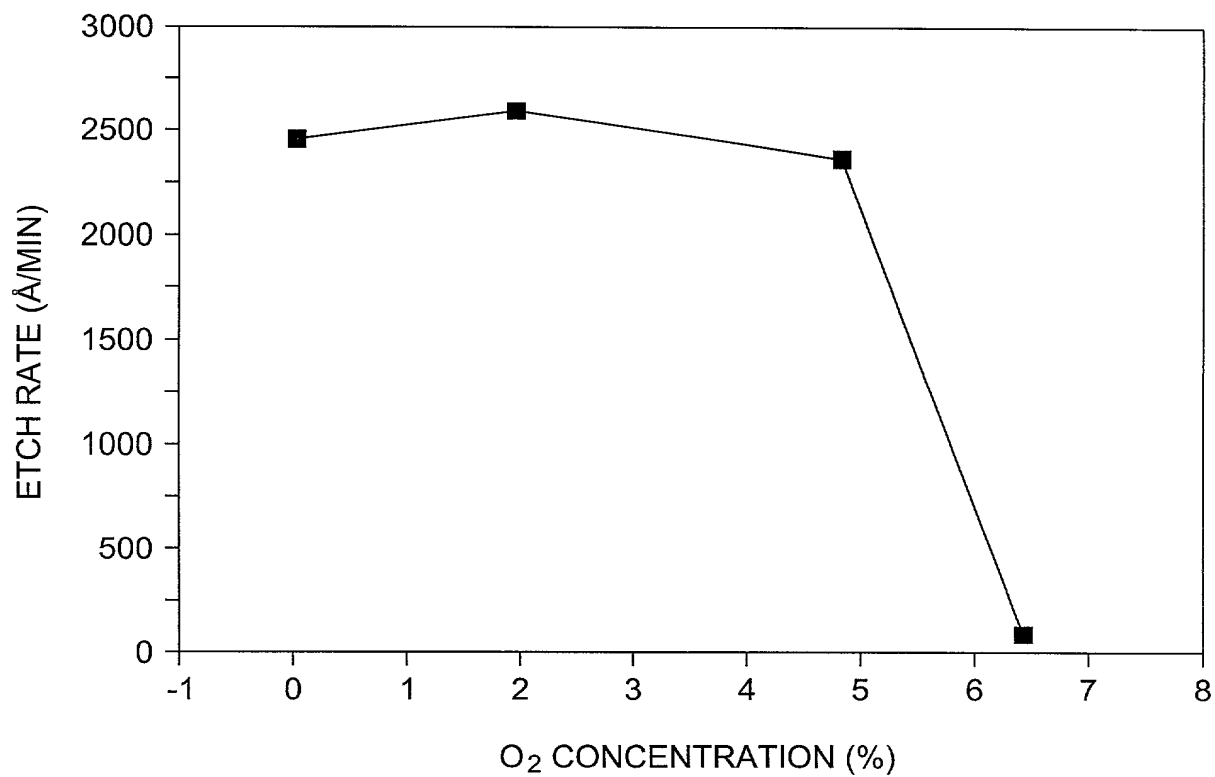
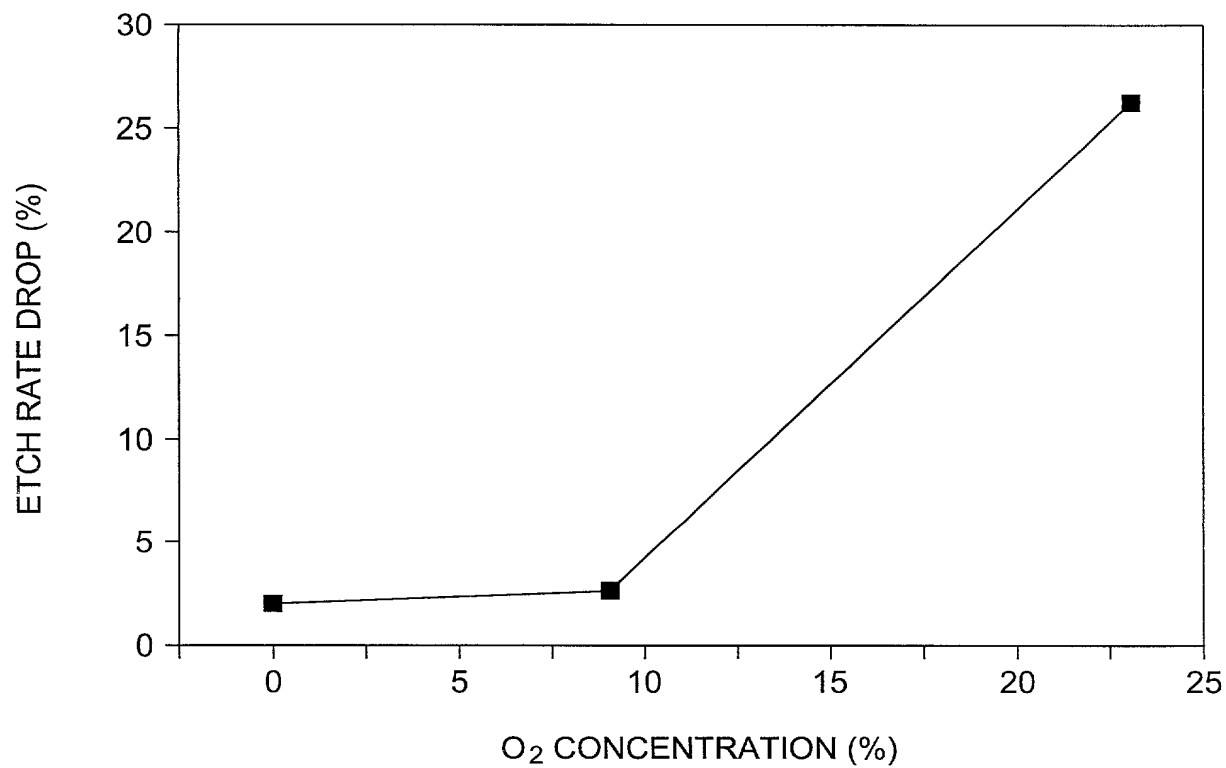


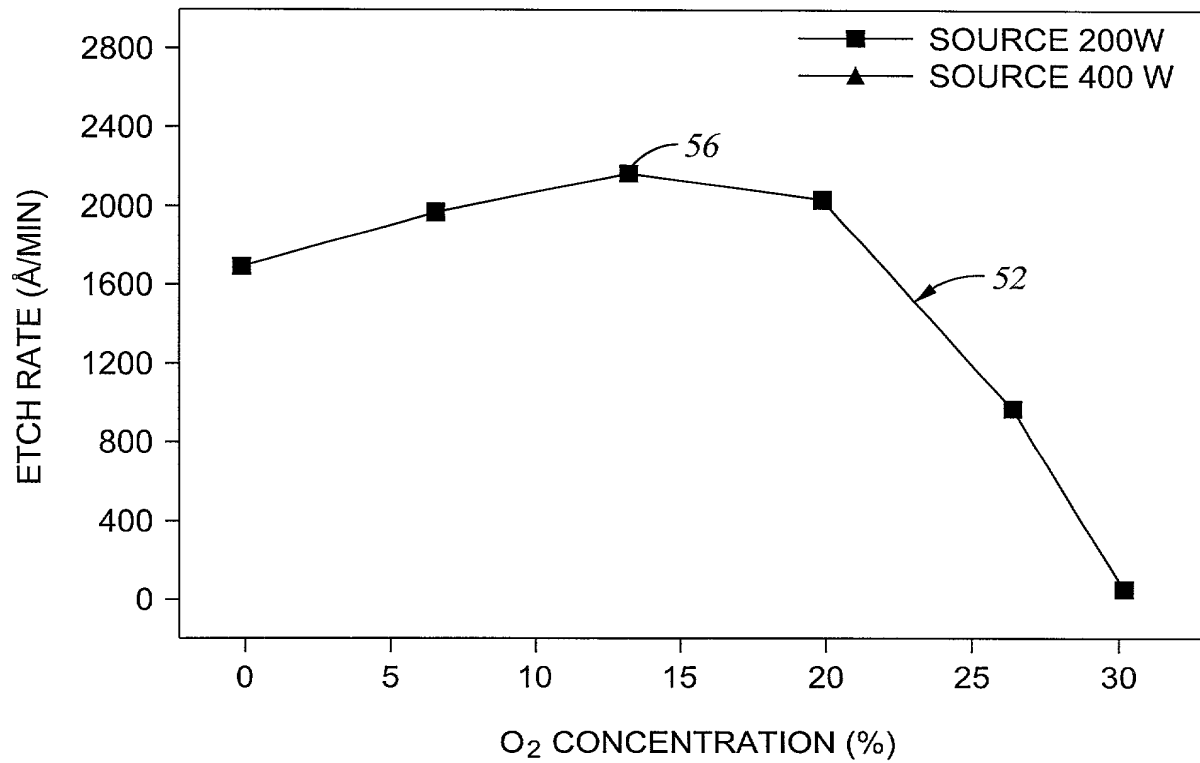
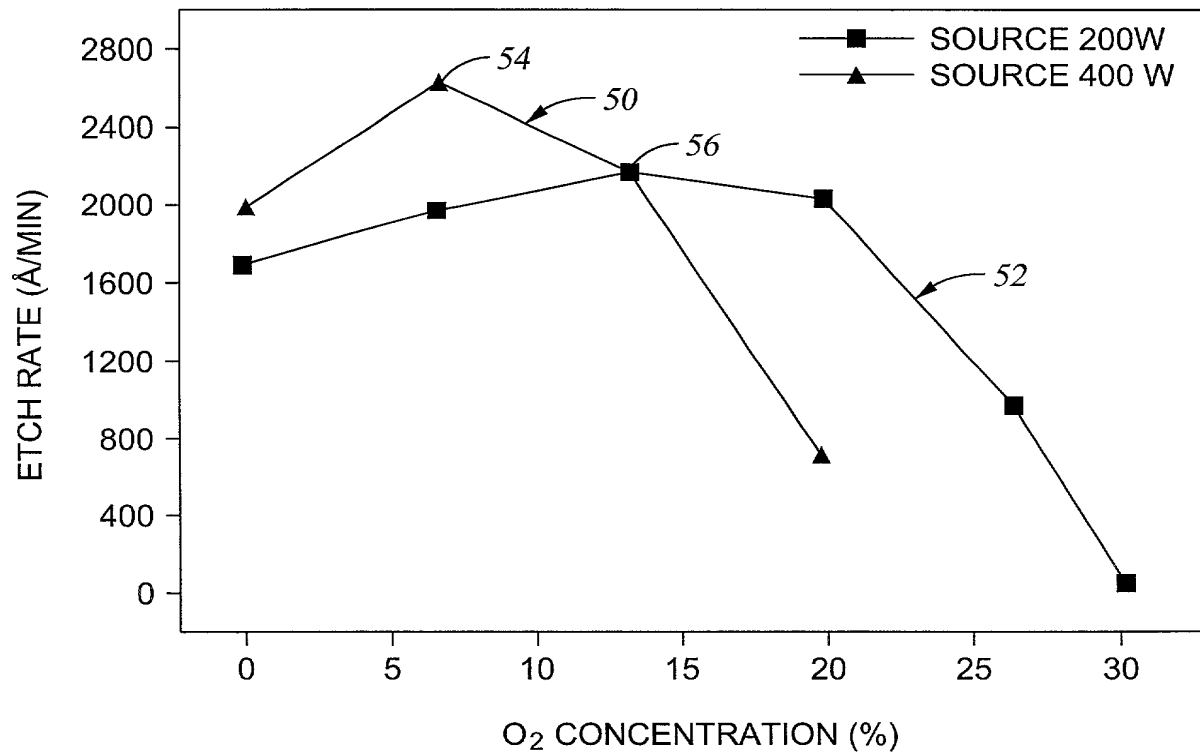
Fig. 1

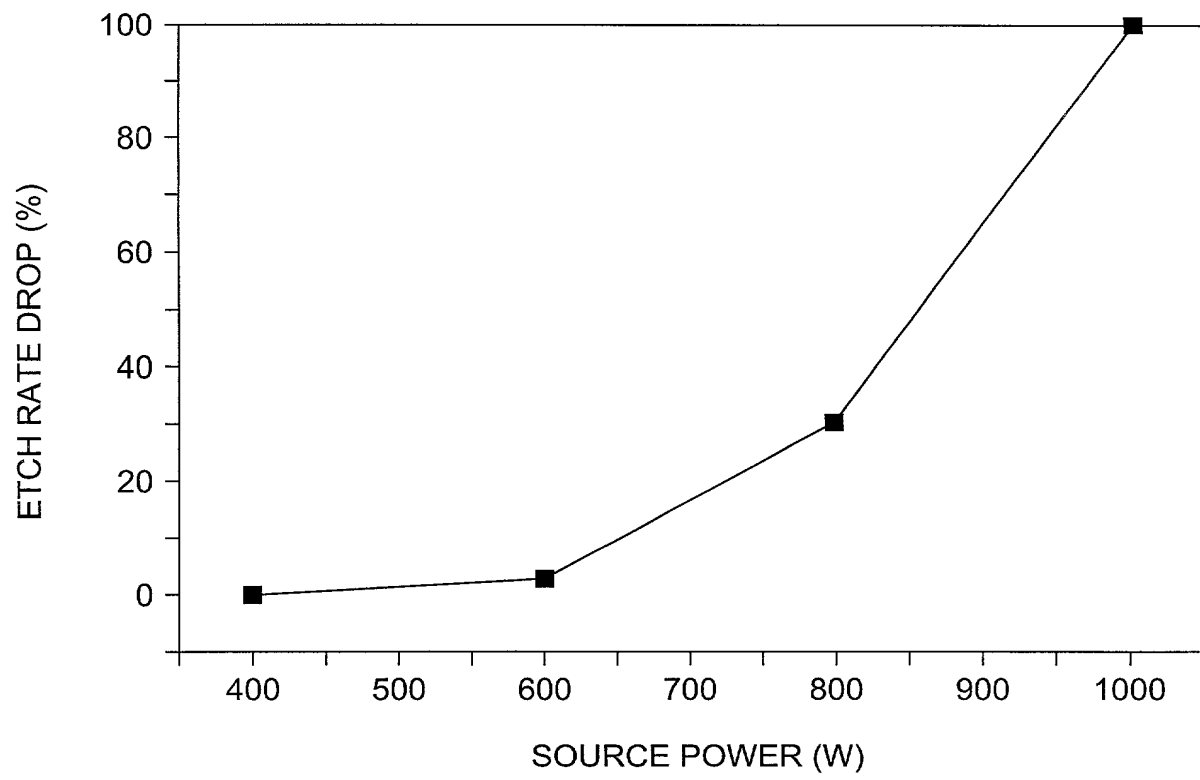
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*Fig. 2**Fig. 3*

*Fig. 4**Fig. 5*

*Fig. 6**Fig. 7*

*Fig. 8**Fig. 9*

*Fig. 10*

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

This declaration is of the following type:

- ☒ original
- ☐ divisional
- ☐ continuation
- ☐ continuation-in-part

INVENTORSHIP IDENTIFICATION

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**NEW METHODOLOGIES TO REDUCE PROCESS SENSITIVITY
TO THE CHAMBER CONDITION**

SPECIFICATION IDENTIFICATION

The specification of which:

- ☒ is attached hereto
- ☐ was filed on _____, under Serial No. _____, executed on even date herewith; or
- ☐ Express Mail No.(as Serial No. not yet known)
- and was amended on _____ (if applicable)
- ☐ was described and claimed in PCT International Application No. _____
- filed on _____ and as amended under PCT Article 19 on _____.

ACKNOWLEDGMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose all information I know to be material to patentability in accordance with Title 37, Code of Federal Regulations, §1.56,

and which is material to the examination of this application; namely, information where there is a substantial likelihood that a reasonable Examiner would consider it important in deciding whether to allow the application to issue as a patent, and

- ☒ In compliance with this duty there is attached an Information Disclosure Statement in accordance with 37 CFR §1.98.

PRIORITY CLAIM (35 U.S.C. §119)

I hereby claim foreign priority benefits under Title 35, United States Code, §119, of any provisional or foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below, and have also identified below any provisional or foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

☒ No such applications have been filed.

☐ Such applications have been filed as follows:

- A. Prior foreign/PCT application(s) filed within 12 mos. (6 mos. for design) prior to this application, and any priority claims under 35 U.S.C. §119

<u>Country/PCT</u>	<u>Application No</u>	<u>Date Filed</u>	<u>Priority Claimed</u>
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No

- B. All foreign application(s), if any, filed more than 12 mos. (6 mos for design) prior to this U.S. application

Country:
Application No:
Filing date:

- C. U.S. Provisional Application filed within 12 months prior to this application

<u>Serial No.</u>	<u>Filing Date</u>
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PRIORITY CLAIM (35 U.S.C. §120)

I hereby claim the benefit under Title 35, United States Code, §120, of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose information that is material to the examination of this application (namely, information where there is substantial likelihood that a reasonable Examiner would consider it important in deciding whether to allow the application to issue as a patent) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application.

☒ No such applications have been filed

☐ Such applications have been filed, as follows:

<u>Serial No.</u>	<u>Filing Date</u>	<u>Status</u>		
		<u>Patented</u>	<u>Pending</u>	<u>Abandoned</u>

POWER OF ATTORNEY

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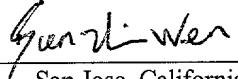
DECLARATION

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Sec. 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patents issued thereon.

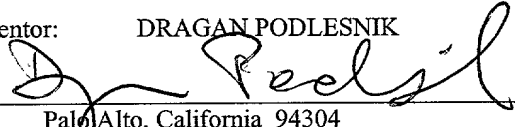
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Inventor's signature: 

Date: 7/9/99

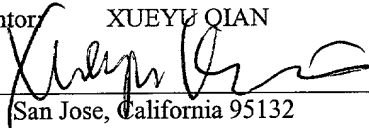
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Country of Citizenship: CHINA

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